UNIVERSITY OF PENNSYLVANIA

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REMBRANDT DEWEES SUMMERS

A DISSERTATION

IN PHYSICS

PRESENTED TO THE FACULTY OF THE GRADUATE SCHOOL IN

PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR

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The Optical Constants of Polished and Sputtered Molybdenum Surfaces

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The optical constants of molybdenum are determined by Drude's method for opaque films sputtered in air and in hydrogen, and for mirrors of massive metal, at wavelengths 0.578μ, 0.546μ and 0.436μ. The indices of refraction and absorption, and the computed reflectivity are found to vary with the conditions of sputtering, and to be considerably less than for the massive metal. To eliminate any possible effect due to surface films formed by chemical action of the atmosphere, the final stage of polishing one of the bulk metal mirrors is carried out under kerosene, and the mirror is observed while immersed in kerosene. The results are not appreciably different when the same mirror is observed in air.

PLANE polarized light reflected from a metallic mirror becomes, in general, elliptically polarized. By observation of this elliptically polarized light, two characteristic constants of the mirror, the index of refraction n and the index of absorption κ may be determined. This method was applied to mirrors of polished and of sputtered molybdenum.

At first a mercury "Lab-Arc" was used as source. For greater intensity, this was later replaced by an air-cooled capillary mercury arc.[1] After passage through a constant deviation monochromator, the light was plane polarized by a Nicol, set successively in the four positions for which the plane of polarization made an angle of 45° with the plane of incidence. The light was then reflected by the molybdenum surface. A Babinet-Soleil compensator restored the resulting elliptically polarized light to plane polarized light whose azimuth was determined by an analyzing Nicol equipped with a Zehnder half-shade.[2] Each set of observations depended on 64 matches of the half-shade field.

The quantities observed were the angle of incidence, the phase change introduced by the compensator, and the azimuth of restored plane polarization. Substitution of these values into Drude's formulas[3] yielded n, κ and the reflectivity R. In this system of notation the complex refractive index is $n[1-(-1)^{\frac{1}{2}}\kappa]$.

PREPARATION OF MIRRORS

(a) Sputtered mirrors

The sputtering chamber was a one gallon jar, arranged as described by Cartwright.[4] The surfaces to receive the sputtered molybdenum rested on a glass table 3 cm below the cathode, and about 50 cm from the aluminum anode. A small 5000 volt "luminous tube" transformer main-

[1] A. Allen and E. Ponder, J. Physiology **66**, 37 (1928).

[2] Zehnder, Ann. d. Physik **26**, 985 (1908).

[3] P. Drude, *Lehrbuch der Optik*, 2nd edition, p. 344.

[4] C. H. Cartwright, R. S. I. **1**, 759 (1930).

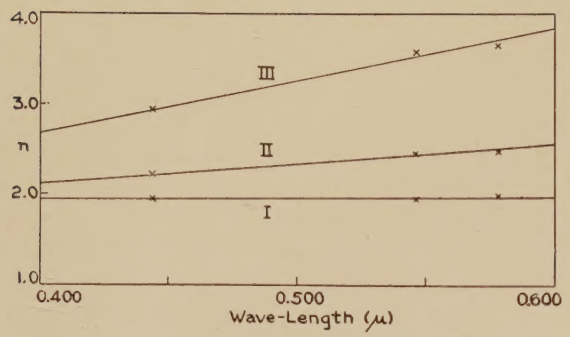

FIG. 1. Index of refraction n plotted against wave-length. Curve I. Molybdenum surface sputtered in hydrogen. Curve II. Molybdenum surface sputtered in air. Curve III. Surface of massive molybdenum.

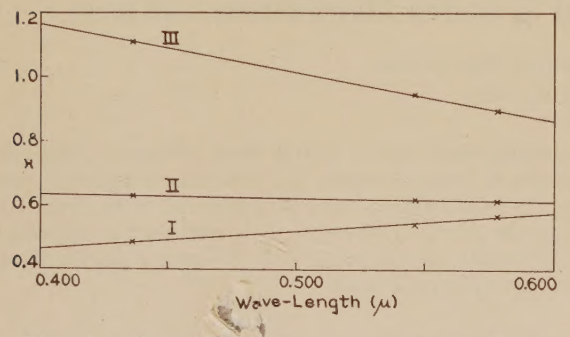

FIG. 2. Index of absorption κ plotted against wave-length. Curve I. Molybdenum surface sputtered in hydrogen. Curve II. Molybdenum surface sputtered in air. Curve III. Surface of massive molybdenum.

tained the gaseous discharge. No rectifier was used.

Three mirrors were sputtered on glass with air as the residual gas. To minimize oxidation the pressure was maintained just above that value at which the discharge ceased to pass. Opaque mirrors were obtained in 13 hours.

Two mirrors were sputtered on glass in electrolytic hydrogen. The pressure in the cathode chamber was regulated so that the Crookes dark space extended about to the mirrors.[5] Opaque mirrors were obtained in 35 hours.

(b) Mirrors of massive metal

Mirrors were prepared from disks of molybdenum, 1/2″ in diameter and 1/4″ thick, stated by the manufacturer to be 99.9 percent pure. These blanks were rubbed by hand on glass

[5] E. O. Hulburt, R. S. I. 5, 85 (1934).

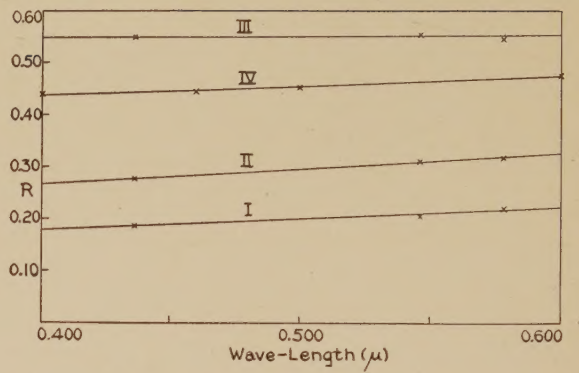

FIG. 3. Reflectivity R plotted against wave-length. Curve I. Molybdenum surface sputtered in hydrogen. Curve II. Molybdenum surface sputtered in air. Curve III. Surface of massive molybdenum. Curve IV. Reflectivity of massive molybdenum by direct measurement. Data from Coblentz.[8]

plates with successively finer grades of carborundum wet with kerosene. They were then buffed on a cotton wheel with occasional application of a paste of optical rouge and water. One mirror was observed without further treatment.

A second mirror was further polished for 2 hours on a rotating lap of beeswax (apiarist's "medium brood foundation" on a plaster base[6]) with rouge and kerosene.

A third mirror, after buffing, was given a brilliant polish by 2 hours' application to a rotating lap of soft cloth, with a thick paste of rouge and water. After a series of optical observations, this mirror was rubbed for 2 hours on a lap of chamois immersed in kerosene, with a very little rouge. It was hoped that this treatment would remove any oxide film which might be present. The mirror was thereafter kept immersed in kerosene. During the observations it was mounted in a kerosene cell with Pyrex windows which were normal, respectively, to the incident and reflected rays. After multiplication of n by the index of refraction of the kerosene, the results agreed within 2 percent for n, and within 1 percent for κ, with the values obtained when the same mirror was observed in air.

The mirrors of massive metal were all found to satisfy Drude's criterion[7] for freedom from contamination: the phase change introduced by

[6] *Amateur Telescope Making*, A. G. Ingalls, Editor, p. 175, Scientific American Pub. Co., 1928.

[7] P. Drude, Ann. d. Physik **39**, 481 (1890).

reflection was as great as for a freshly filed surface.

Results

Table I gives averages computed from 37 sets of observations at angles of incidence between

Table I.

		n	κ	R
Mirrors sputtered in hydrogen				
Wave-length	0.436μ	1.95	0.484	0.187
	0.546μ	1.96	0.542	0.206
	0.578μ	1.99	0.566	0.220
Mirrors sputtered in air				
Wave-length	0.436μ	2.22	0.626	0.278
	0.546μ	2.46	0.619	0.312
	0.578μ	2.48	0.614	0.318
Mirrors of massive metal				
Wave-length	0.436μ	2.95	1.113	0.551
	0.546μ	3.59	0.948	0.556
	0.578μ	3.65	0.897	0.548

45° and 70°. These data are plotted in Figs. 1, 2 and 3. Curve IV, Fig. 3, was plotted from data of Coblentz,[8] obtained by direct measurement of the reflectivity of molybdenum.

[8] W. W. Coblentz, Bur. Standards Bull. **7**, 107 (1911).

The residual gas appears to play an important rôle in the optical constants of the sputtered surfaces. The mirrors sputtered in hydrogen have much lower index of refraction and reflectivity than those mirrors which were sputtered in air. Ingersoll and Hanawalt[9] have shown that, in films of nickel sputtered in hydrogen, the crystal lattice may be distended by occluded gas, with striking effect on the physical properties of the films. It is not unlikely that the optical properties of the sputtered mirrors in this research are altered by this phenomenon. Lauch[10] associates low values of κ with "loose" atomic arrangement, as may be caused by occluded gas. This agrees with the low κ found for sputtered mirrors, and the higher κ for the mirrors of massive metal.

In conclusion the writer desires to express his deep appreciation to Professors H. C. Richards and R. W. Duncan for many helpful suggestions and for their continued interest in this research.

[9] L. R. Ingersoll and J. D. Hanawalt, Phys. Rev. **34**, 973 (1929).

[10] K. Lauch, Ann. d. Physik **74**, [illegible] (1924).

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